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कैसिन आधारित जलीय वर्णक और  
परिसज्जा ( फिनिश ) — विशिष्टि  
रूप — रीति संहिता  
( पहला पुनरीक्षण )

Casein Based Aqueous Pigments  
and Finishes — Specification  
( First Revision )

ICS 59.140.10

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## FOREWORD

This Indian Standard ( First Revision ) was adopted by the Bureau of Indian Standards, after the draft finalized by the Leather, Tanning Materials and Allied Products Sectional Committee had been approved by the Chemical Division Council.

Casein based aqueous pigments are used widely in the country along with casein based binders and synthetic polymeric lattices. The water dispersible pigments, that is, the basic colouring matter in aqueous finishes, are prepared by dispersing dry pigment colours or pulps, such as, synthetic, precipitated inorganic or organic pigments and earth colours in casein solutions with suitable amount of plasticizers and other ingredients, such as, thickeners, to impart desired gloss. To contribute some specific properties to the finish other subsidiary binders are also added. These pigments which are generally in thick viscous paste form are prepared in a variety of colours and shades. The general characteristics like viscosity; total solid; covering power; protein content; preservatives and oils, fats, and waxes used in the manufacture, differ from one manufacturer to another. Some manufacturers market these pigments in a ready-to-use form for direct application to the leather with or without extra casein based binders which are marketed separately for finishing different types of leather, others market pigments and binders separately with their own auxiliary finishing agents to be used in proportions recommended by them. The amounts of the binders and pigments to be used in the finishing of different types of leathers also vary considerably. There are still some consumers who use the commercial pigments and prepare their own binders for use admixed with synthetic or other types of binders.

In all these cases, however, the pigments and binders have to be used essentially in the finishing of different types of leathers used for goods of daily use like shoe uppers, case sides and portfolio leathers. Essentially the finishes, especially the pigment pastes and binders commonly used, shall be of a standard quality. The comparison of two brands of pigment pastes on the basis of the analytical figures will not indicate any idea about the quality of the finish, as it is only the final formulation at the hands of the finisher that mostly decides the quality of the finish. As such, the best way to judge the performance of the finish is to finish the leather as recommended by the manufacturer and then test for the various finishing properties, according to the methods prescribed in this specification.

In view of the complexity of the types of finishes the formulation of this standard has been approached from the practical point of view and stress has been laid on physical and practical tests in preference to chemical tests which fail to give any clue as to the satisfactory nature of the products.

This standard was originally published in 1966. In this revision, the requirement on pentachlorophenol (PCP) has been introduced keeping in view of the demand for eco-friendly inputs from the leather industry. Reference has also been made to the latest test methods.

The composition of the Committee responsible for the formulation of this standard is given in Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# *Indian Standard*

## CASEIN BASED AQUEOUS PIGMENTS AND FINISHES — SPECIFICATION

### ( *First Revision* )

#### 1 SCOPE

**1.1** This standard prescribes the requirements, methods of sampling and test for casein based aqueous pigment finishes and casein based binders with or without dispersed pigments for leather.

**1.2** This standard does not cover the dispersed pigments or concentrated pigment colours which do not contain any casein binder and which are generally meant for use with synthetic resin emulsions or other commercially known synthetic resin binders used in the leather industry.

#### 2 REFERENCES

The standards given below contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of these standards:

<i>IS No.</i>	<i>Title</i>
101	Methods of sampling and test for paints, varnishes and related products:
(Part 1/Sec 1) : 1986	Test on liquid paints (general and physical), Section 1 Sampling
(Part 2/Sec 2) : 1986	Test on liquid paints (chemical examination), Section 2 Volatile matter
(Part 4/Sec 2) : 1989	Optical tests, Section 2 Colour
(Part 4/Sec 4) : 1988	Optical tests, Section 4 Gloss
1640 : 2007	Glossary of terms relating to hides, skins and leather
6191 : 1971	Method of micro-biological colour fastness and microscopical tests for leather
14575 : 1999	Determination of pentachlorophenol (PCP) in leather — Method of test

#### 3 TERMINOLOGY

For the purpose of this standard, the definitions given in IS 1640 shall apply.

#### 4 REQUIREMENTS

##### 4.1 Physical Appearance and Nature of the Product

The pigment paste shall be homogeneous, uniform, of viscous consistency, smooth to touch when rubbed between two fingers or should have no undispersed or agglomerated particles, caking of the pigment in the container and shall be easily dispersible when mixed with water. On storage there shall be no sedimentation that shall not be readily dispersed by gentle stirring or shaking.

##### 4.2 Uniformity

When tested as prescribed in IS 101 (Part 2/Sec 2) and IS 101 (Part 4/Sec 2) for volatile matter and colour, all the lots in a single consignment shall be as agreed to between the purchaser and the supplier.

**4.3** The particle size of the pigment dispersion shall be of the order of 2.5 to 5  $\mu\text{m}$  with exception of a few stray agglomerates not exceeding 25  $\mu\text{m}$ , when determined using a fineness meter or when tested under a microscope.

**4.4** The casein binder shall be homogeneous, uniform in consistency without showing any signs of sedimentation, and shall be opalescent. The film dried from the binder shall be clear.

**4.4.1** The casein binder shall have a pH range 6 to 8.5 on dilution with five times its water, determined using a pH meter.

**4.5** During the manufacture of the binder and the pigment paste, necessary quantities of preservative for the casein may be incorporated so as to ensure a shelf life of at least one year, if stored in closed containers in which the material had been originally received by the purchaser and stored in cool and dry place.

##### 4.5.1 Freedom from Pentachlorophenol (PCP)

The material shall not contain more than 5 mg/kg of pentachlorophenol when tested in accordance with Annex A.

**4.6** The shade and the covering power shall be as agreed to between the purchaser and the supplier.

4.7 The pigment finish shall be applied in accordance with the manufacturer's instructions to test pieces of dyed crust leather and shall be tested for the various finishing properties as given in Table 1.

## 5 PACKING AND MARKING

### 5.1 Packing

The material after manufacture shall be packed in tight containers which shall not develop any rust or deterioration of the product due to the presence of water in the medium or any of the ingredients used in the formulation.

### 5.2 Marking

Each container shall be marked with the following:

- Name of the material and shade;
- Name of the manufacturer and/or trade-mark, if any;
- Volume of the material;
- Batch number;
- Month and year of manufacture; and

- Instructions for preparing the leather finishing formulation.

### 5.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard mark is governed by the provisions of the *Bureau of Indian Standard Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## 6 SAMPLING AND CRITERIA FOR CONFORMITY

### 6.1 Scale of Sampling

For the purpose of ascertaining the conformity of the material in any consignment to this specification, the scale of sampling and criteria for conformity shall be as prescribed in 3 of IS 101 (Part 1/Sec 1).

**Table 1 Requirements for Finishing Properties of Aqueous Casein Pigments and Finishes**  
(Clauses 4.7, B-1.1, B-4 and B-5)

Sl No.	Characteristic	Requirement	Methods of Test, Ref to	
			Clause No. of IS 6191 (4)	Clause No. of Annex B (5)
(1)	(2)	(3)		
i)	Colour fastness test for finished leather (contrast grading after 1024 revolutions), not worse than:		LF 9	—
	a) Dry rubbing	4		
	b) Wet rubbing	3		
ii)	Gloss of the finished leather	Shall have the same degree of gloss as agreed to between the purchaser and the supplier	—	B-2
iii)	Resistance to lasting	To pass test	—	B-3
iv)	Fastness to day light	3-4	LF 3	—
v)	Fastness to organic solvents	The finish shall not crack, peel or crumble	LF 6	—
vi)	Fastness to water spotting	There shall be no swelling, loss of luster or any visible change when compared with the original spot	LF 8	—
vii)	Resistance to heat	To pass test	—	B-4
viii)	Ageing	To pass test	—	B-5

## ANNEX A

(Clause 4.5.1)

### DETERMINATION OF PENTACHLOROPHENOL (PCP)

**A-1** Transfer about 5 g of the casein based aqueous pigment or finish into a separating funnel of about 250- ml capacity along with 100 ml water. Add 15-25 ml of sulphuric acid 10 percent w/v aqueous solution and ensure that the pH of the contents is acidic. Then carry out liquid — liquid extraction using hexane, analytical grade, as the solvent. At first extract with about 25 ml hexane by shaking the contents thoroughly for about 15 min. Then set it aside for clear separation of the layers (*see* Note). Transfer the aqueous layer into another separating funnel and carry out three more extractions of the

aqueous layer with hexane of 25 ml each time in the same manner explained earlier.

NOTE — In case of any emulsion being noticed, break the same with the addition of 20-30 ml saturated sodium chloride aqueous solution or follow the steps explained in 4.3.4.2 of IS 14575.

**A-2** Combine the hexane extracts and dry it by passing through a bed of anhydrous sodium sulphate of about 10 g. The dried hexane layer is evaporated to near dryness by a rotary evaporator.

**A-3** The residue is taken for acetylation. Then follow the procedure as detailed in Method A of IS 14575.

## ANNEX B

(Table 1)

### TESTS ON AQUEOUS CASEIN BASED PIGMENTS AND FINISHES

#### B-1 GENERAL

**B-1.1** The samples of the aqueous casein based pigment or finishes or both shall be tested for finishing properties (*see* Table 1) by application on dyed crust leather pieces, of size 30 cm × 30 cm. At least three test pieces shall be taken for each test. The pigments shall be tested using a casein binder to be supplied by the manufacturer or prepared according to the recipe, furnished by the manufacturer.

**B-1.2** The leather pieces shall be finished as per detailed method to be furnished by the manufacturer using the auxiliaries as suggested by him. All the ingredients shall be of the quality as agreed to between the purchaser and the supplier, wherever such requirements are not prescribed for the ingredients in this standard.

#### B-2 Gloss of the Finished Leather

The gloss of the finished leather when tested as prescribed in IS 101 (Part 4/Sec 4) shall be as agreed to between the purchaser and the supplier.

#### B-3 Resistance to Lasting

A key drawn against the leather from the flesh side by holding the leather firm against key between the thumb and the first finger shall show no signs of crackiness or peeling of pigment film. The finished leather shall show no sign of crack, peel or crumble of the pigment film on double fold.

#### B-4 Resistance to Heat

The leather, after the application of the finish shall be hot plated or ironed. The finished surface shall comply with the requirements of Sl. No. (i), (iii), (v) and (vi) of Table 1 after the hot plating or ironing.

#### B-5 Ageing Test

The leather, after the application of the finish shall be hot plated or ironed. The test piece shall then be kept for 1 h in an air oven at  $70 \pm 1^\circ\text{C}$  and subsequently cooled to room temperature. The test piece shall then be checked for compliance to Sl No. (i), (iv), (v) and (vi) of Table 1.

## ANNEX C

### (Foreword)

#### COMMITTEE COMPOSITION

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### Amendments Issued Since Publication

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